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Indian Standard

SPECIFICATION FOR PHENYL PERI ACID, TECHNICAL

(First Revision)

UDC 667·289 : 547·661·1·269·3

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 3 *August* 1987

AMENDMENT NO. 1 AUGUST 2002
TO
IS 8233 : 1987 SPECIFICATION FOR PHENYL PERI ACID, TECHNICAL

(First Revision)

(*Page* 3, *clause* **0.2**, *Structural Formula*) — Insert the following below the structural formula:

'(CAS No. 82-76-8).'

[*Page* 4, *Table* 1, *Sl No* (iv)] — Substitute the following for the existing:

(1)	(2)	(3)	(4)
iv)	Matter insoluble in sodium carbonate solution, percent by mass, *Max*	0 3	when tested as per IS 5299 2001

(PCD 11)

Reprography Unit, BIS, New Delhi, India

Indian Standard

SPECIFICATION FOR PHENYL PERI ACID, TECHNICAL

(First Revision)

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(*Continued on page* 2)

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Indian Standard

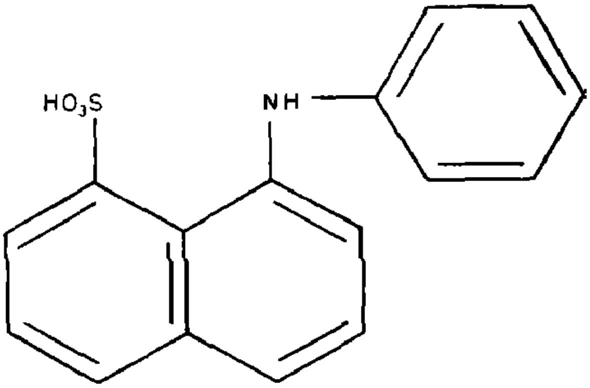
SPECIFICATION FOR
PHENYL PERI ACID, TECHNICAL

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 2 January 1987, after the draft finalized by the Dye Intermediates Sectional Committee had been appproved by the Petroleum, Coal and Related Products Division Council.

0.2 Phenyl peri acid ($C_{16}H_{13}O_3NS$) is an important intermediate used in the manufacture of dyes. It is also known as 1-phenyl aminonaphthalene-8-sulphonic acid. It is available as a free acid or its salts like sodium, magnesium or ammonium. It is represented by the following structural formula:



PHENYL PERI ACID
(Molecular Mass 299)

0.3 This standard was first issued in 1976. The committee responsible for the preparation of this standard decided to undertake its revision in order to modify the requirement of peri acid content and its method of determination, and also to incorporate the requirement of aniline content by thin layer chromatographic method.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 *This standard prescribes the requirements, and the methods of* sampling and test for phenyl peri acid, technical.

2. REQUIREMENTS

2.1 Description — The material shall be either free acid or in the form of its salt like sodium, magnesium or ammonium. It shall be in the form of a paste or, if dry, in the form of grey to greenish grey lumps or powder.

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR PHENYL PERI ACID, TECHNICAL

(*Clauses* 2 2, 4.3 1, 4 3.2 *and* 5 1)

SL No	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO CL No. IN APPENDIX A
(1)	(2)	(3)	(4)
i)	Assay, percent by mass (on dry basis), *Min*	80 0	A-2
ii)	Peri acid content, percent by mass (on dry basis), *Max*	1·0	A-3
iii)	Aniline content, percent by mass (on dry basis), *Max*	0 2	A-4
iv)	Solubility in sodium carbonate solution*	To pass test	A-5

*This requirement is not applicable to magnesium salt.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in steel drums (*see* IS : 2552-1979†) lined with suitable polyethylene film or as agreed to between the purchaser and the supplier.

---

*Rules for rounding off numercial values (*revised*).

†Specification for steel drums (galvanized and ungalvanized) (*second revision*).

3.2 Marking — Each container shall be securely closed and shall bear legibly and indelibly the following information:

a) Name of the material;

b) Name of the manufacturer and his recognized trade-mark, if any;

c) Gross, net and tare mass;

d) Batch number; and

e) Minimum cautionary notice worded as under:

'IT IS A MILD SENSITIZER. LOCAL CONTACT MAY CAUSE DERMATITIS'.

3.2.1 The containers may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers, may be obtained from the Bureau of Indian Standards.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in **3** of IS : 5299-1969*.

4.2 Number of Tests

4.2.1 Test for assay shall be conducted on each of the individual samples

4.2.2 Tests for the determination of remaining characteristics, namely, peri acid and aniline content, and solubility in sodium carbonate solution shall be conducted on the composite sample.

4.3 Criteria for Conformity

4.3.1 *For Individual Samples* — The lot shall be declared as conforming to the requirement of assay if each of the individual test results satisfies the relevant requirement given in Table 1.

4.3.2 *For Composite Sample* — For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample (*see* **4.2.2**), the test results for each of the characteristics shall satisfy the relevant requirements given in Table 1

---

*Methods for sampling and tests for dye intermediates.

5. TEST METHODS

5.1 Tests shall be conducted according to the methods prescribed in Appendix A. Reference to relevant clauses of Appendix A is given in col 4 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

(*Table* 1 and *Clause* 5.1)

METHODS OF TEST FOR PHENYL PERI ACID, TECHNICAL

A-1. PREPARED SAMPLE

A-1.1 Dry the material at 105 ± 2°C in an oven to constant mass. Grind and mix well. Transfer the material to a wide-mouthed bottle and stopper it. Do not expose the sample to an atmosphere containing acidic or alkaline fumes. Use this *prepared sample* for the tests.

A-2. ASSAY

A-2.0 Outline of the Method — The material is dissolved in dilute sodium carbonate solution A known volume of the solution is coupled with standard 4-chloroaniline diazo in sodium acetate medium and from the consumption of diazo, strength is calculated.

A-2.1 Reagents

A-2.1.1 *Sodium Carbonate Solution* — Approximately 10 percent (*m/v*).

A-2.1.2 *Phenolphthalein Indicator Paper*

A-2.1.3 *Standard 4-Chloroaniline Diazo Solution* — 0·1 N.

A-2.1.4 *H-acid Indicator* — neutral

A-2.1.5 *Sodium Acetate Solution* — 30 percent (*m/v*).

---

*Specification for water for general laboratory use (*second revision*).

A-2.2 Procedure — Weigh accurately about 15 g of the *prepared sample* (*see* **A-1.1**) and transfer to a 500-ml beaker with the help of water. Slowly add sodium carbonate solution to get a positive test on phenolphthalein indicator paper. Add about 300 ml of warm water and stir at 60 to 70°C to dissolve the material. Filter the solution and wash the residue with hot water. Transfer quantitatively the filtrate and washings into a 500-ml volumetric flask and dilute up to the mark with water at room temperature. Mix well. Pipette 50 ml aliquot of this solution into a 1-litre beaker. Add 200 ml of ice-cold water and 175 ml of sodium acetate solution. Stir mechanically and cool with washed ice to 0·5°C. Titrate with 4-chloroaniline diazo solution from a cold water-jacketted burette. Test with H-acid for the excess diazo and with diazo for the coupling component. The end-point is noted when there is no test with 4-chloroaniline diazo and a fine visible pink line with H-acid indicator is obtained which can persist for 10 minutes without further addition of diazo

NOTE — The coupling towards the end is slow and may give tests with both the indicators Allow to react for sometime and proceed further Near the end-point, add the diazo in 0·5 ml portions

A-2.3 Calculation

$$\text{Assay, percent by mass (on dry basis)} = \frac{V \times N \times 29{\cdot}9}{M}$$

where

V = volume in ml of the standard 4-chloroaniline diazo solution,

M = mass in g of the material taken for the test, and

N = normality of 4-chloroaniline diazo solution.

A-3. PERI ACID CONTENT

A-3.0 Outline of the Method — Peri acid is estimated by descending paper chromatographic technique.

A-3.1 Reagents

A-3.1.1 *Phenyl Peri Acid* — Free from peri acid.

A-3.1.2 *Peri Acid* — Pure (reference sample).

A-3.1.3 *Ammonium Hydroxide Solution* — 1·0 percent (*m/v*).

A-3.1.4 *Mobile Phase* — *n*-butanol saturated with water.

A-3.2 Apparatus

A-3.2.1 *Developing Glass Chamber for Descending Chromatography*

A-3.2.2 *Chromatographic Sprayer*

A-3.2.3 *Micro-Pipette* — 10 μl capacity.

A-3.2.4 *UV lamp*

A-3.3 Procedure

A-3.3.1 Prepare standard solution of phenyl peri acid containing known amounts of peri acid in the following manner.

A-3.3.1.1 Into each of three 100-ml volumetric flasks, weigh accurately 1·0 g of phenyl peri acid (**A-3.1.1**). Then add 1 ml, 2 ml and 3 ml of 0·5 percent solution of peri acid (**A-3.1.2**) in ammonium hydroxide solution to flasks No. 1, 2 and 3 respectively. Dissolve the contents of the flask in ammonium hydroxide solution Then make the volume up to the mark with ammonium hydroxide solution. Thus there are three solutions of 0 5, 1·0 and 1·5 percent peri acid content.

A-3.3.1.2 In a fourth flask, weigh accurately 1·0 g of the *prepared sample* (*see* **A-1.1**), dissolve in ammonium hydroxide solution and dilute to 100 ml mark with ammonium hydroxide solution.

A-3.3.1.3 Place 10 μl spot of each of the four solutions using micropipette on the same line to a distance of about 4 cm on Whatman No. 1 filter paper. Place the paper to a descending paper chromatographic glass jar containing the developing reagent and previously saturated with the same developer Allow the solvent to run in a descending manner for about 20 cm from the spot. This will take about 7 hours. Maintain the temperature of the chamber at room temperature Take out the paper after 20 cm run and dry the solvent completely. The chromatogram so developed is observed under UV light when the separately spots characteristic of constituents may be identified by their colours as under:

Content	*Zone*	*Rf Value*	*Colour*
Unknown	I	0 13	Faint brown
Peri acid	II	0·49	Greenish
Phenyl peri acid	III	0 73	Greenish

A-3.4 Report — Report peri acid content as that which is nearest in intensity to the standard In case the colour intensity does not come in the range of the standard spots, repeat the whole procedure using different percentage of peri acid.

A-4. ANILINE CONTENT

A-4.0 Outline of the Method — Aniline is estimated by ascending thin layer chromatographic technique.

A-4.1 Reagents

A-4.1.1 *Aniline* — pure.

A-4.1.2 *Phenyl Peri Acid* — free from aniline.

A-4.2.3 *Methanol Solution.*

A-4.1.4 *Mobile Phase* — Benzene.

A-4.1.5 *Diazotizing Solution* — Dissolve one part of 1 N sodium nitrite solution in two parts of 1 N hydrochloric acid.

A-4.1.6 *Coupling Solution* — Dissolve 0·5 g of α-naphthol in 20 ml of ethyl alcohol and 80 ml of 10 percent (*m/v*) sodium carbonate solution.

A-4.1.7 *Silica Gel* — neutral.

A-4.2 Apparatus

A-4.2.1 *Chromatographic Chamber* — 22 × 10 × 22 cm.

A-4.2.2 *Thin Layer Chromatographic Glass Plates* — 20 × 20 cm.

A-4.2.3 *Adjustable Thin Layer Applicator.*

A-4.2.4 *Micropipette* — 10 µl capacity.

A-4.2.5 *Chromatographic Spray Bottle*

A-4.2.6 *Oven* — with a thermoregulator.

A-4.2.7 *Weighing Bottle*

A-4.3 Procedure

A-4.3.1 *Preparation of the Chromatographic Plates* — Mix 50 g of neutral silica gel with 85 to 90 ml of water for three minutes. Then, by means of the applicator, coat the plate with the silica gel layer of thickness not exceeding 0·25 mm and place it in the oven maintained at 110°C for 90 minutes By means of a needle, mark a thin line widthwise on all plates at about 3 cm from the upper edge Also remove a band of about 5 mm width silica gel from either of the lateral sides of each plate.

A-4.3.2 *Preparation of Sample Solution*

A-4.3.2.1 Prepare standard solution of phenyl peri acid containing known amounts of aniline in the following manner.

Into each of three 100-ml volumetric flasks, weigh accurately 1 0 g of phenyl peri acid (**A-4.1.2**). Then add 0 5 ml, 1 ml and 1·5 ml of 0·2 percent solution of aniline (**A-4.1.1**) in methanol to flasks No. 1, 2 and 3 respectively. Dissolve the contents of the flasks in methanol and make up to the mark. Thus there are three solutions of 0 1, 0·2 and 0 3 percent aniline content.

A-4.3.2.2 In the fourth flask, weigh accurately 1 0 g of the *prepared sample* (*see* **A-1.1**), dissolve in methanol and dilute to 100 ml mark with methanol.

A-4.3.3 Spot 10 μl each of the reference solutions (**A-4.3.2.1**) and sample solution (**A-4.3.2.2**) on the thin layer plate. Place the developer in the developing chamber. Close the chamber with its lid and allow to achieve equilibrium. Now place the plate carefully in the chamber and allow the mobile phase to run in the ascending manner to about 13 cm. This will take about one hour. Remove the plate from the chamber and dry off the solvent in an air drier. Hold the plate for 20 seconds in nitrous acid fumes formed by adding sodium nitrite and hydrochloric acid (**A-4.1.5**) Allow the plate to stand for 1 minute to remove excess nitrous fumes. Spray with coupling solution (**A-4.1.6**). The chromatogram so developed is visually observed when the separated spots characteristic of constituents may be identified by their colour as under:

Content	*Zone*	*Ref Value*	*Colour*
Main	I	0·00	Greenish yellow
Aniline	II	0·35	Reddish brown

A-4.4 Report — Report aniline content as that which is nearest in intensity to the standard. In case the colour intensity does not come in the range of the standard spots, repeat the whole procedure using different percentage of aniline.

A-5. SOLUBILITY IN SODIUM CARBONATE SOLUTION

A-5.1 Reagent

A-5.1.1 *Sodium Carbonate Solution* — Approximately 5 percent (*m/v*).

A-5.2 Procedure

A-5.2.1 Weigh about 10 g of the *prepared sample* (*see* **A-1.1**) in a 250-ml beaker. Add 100 ml of sodium carbonate solution and heat to dissolve the material completely.

A-5.2.2 The material shall be taken to have passed the test if the solution remains clear and dark-brown in colour.

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